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(54) [Title of the Invention] Solid-Liquid Separation Method

(57) [Summary]

[Object] To provide a solid-liquid separation method that has been improved so that clogging is prevented in a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation can be performed efficiently.

[Means of Achievement] A slurry containing particles of indeterminate form as solids is continuously supplied to a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation is performed, wherein a screen with an opening size that allows the passage of an amount of solids equivalent to 1 to 10 wt% in the supplied slurry is used as the screen of the screen-type centrifugal separator.

[Claims]

[Claim 1] A solid-liquid separation method, characterized in that a slurry containing particles of indeterminate form as solids is continuously supplied to a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation is performed, wherein a screen with an opening size that allows the passage of an amount of solids equivalent to 1 to 10 wt% in the supplied slurry is used as the screen of the screen-type centrifugal separator.

[Claim 2] The solid-liquid separation method according to claim 1, wherein the screen-type centrifugal separator has an outer rotating cylinder (1), a screw conveyer (2) that is mounted inside the outer rotating cylinder while allowed to rotate in relative fashion and that comprises a cylindrical rotating shaft (21) and a screw (22), and a slurry supply pipe (3) that is disposed within the rotating shaft of the screw conveyer and that supplies a slurry to the interior of the shaft; a slurry supply port (23) for supplying a slurry to the outer rotating cylinder (1) is provided to the proximal end side of the screw conveyer (2); the outer rotating cylinder (1) is sequentially constituted from a large-diameter region (11) on the proximal end side, a slanted region (12) of gradually decreasing diameter, and a small-diameter region (13) on which a screen (13a) is provided; an overflow port (4) is provided to the proximal end of the large-diameter region (11); and a solids discharge port (5) is provided to the distal end of the small-diameter region (13).

[Claim 3] The solid-liquid separation method according to claim 2, wherein the interior of the rotating shaft (21) of the screw conveyer is partitioned into a slurry supply region (21a) on the proximal end side and a cleaning fluid supply region (21b) on the distal end side, with the slurry supply region (21a) designed to be capable of supplying the slurry via the slurry supply pipe (3), and the cleaning fluid supply region (21b) designed to be capable of supplying the cleaning fluid via a cleaning fluid supply pipe (6) inserted into the interior of the slurry supply region (3); and a cleaning fluid supply port (24) through which cleaning fluid is supplied to the small diameter region (13) of the outer rotating cylinder (1) is provided to the distal end side of the screw conveyer (2).

[Detailed Description of the Invention]

[0001]

[Technological Field of the Invention] The present invention relates to a solid-liquid separation method, and specifically relates to a solid-liquid separation method that uses a screen-type centrifugal separator with an internally disposed screw conveyor.

[0002]

[Prior Art] In steps for manufacturing terephthalic acid, e.g., by means of subjecting *p*-xylene to liquid-phase oxidation, the terephthalic acid precipitates as crystals, and a slurry is formed. The solids (terephthalic acid crystals) obtained as a result of such crystallization are particles of indeterminate form with a particle size distribution.

[0003] One method of subjecting slurries containing such particles of indeterminate form as described above to solid-liquid separation involves continuously supplying the slurry to a screen-type centrifugal separator with an internally disposed screw conveyor, whereupon solid-liquid separation occurs on the screen due to the application of centrifugal force.

[0004] When a screen is used to perform solid-liquid separation, it is important for the screen to be prevented from getting clogged so that separation can be performed efficiently. Clogging is especially exacerbated in screen-type centrifugal separators with screw conveyors as a result of solidified (cake) layers that form in a compacted state on the inside of the screen.

[0005] JP (Kokai) 8-294643 proposes a scraper for scraping off the solidified layers in the screen to maintain its function, but a problem is presented insofar as the system becomes complicated due to the presence of the scraper. Furthermore, as relates to the size of the screen openings, it is stated in e.g. "Filtration & Separation (Sep., 2000, p. 245)" that the size of the screen openings should preferably be set to be two to three times the particle size of the solid to be separated in order to prevent the particles from becoming lodged in the screen and clogging it. Nevertheless, such large-mesh screens have the problem of allowing too much of the material to pass through the screen openings.

[0006]

[Problems to Be Solved by the Invention] With the foregoing aspects in view, it is an object of the present invention to provide a solid-liquid separation method that has been improved so that

clogging is prevented in a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation can be performed efficiently.

[0007]

[Means Used to Solve the Above-Mentioned Problems] In order to achieve the above objectives, the present inventors conducted diligent investigations into the solidified (cake) layers formed in a compacted state by the screw on the inside of the screen, and obtained the findings hereunder as a result. In other words, if particles of indeterminate form and relatively small size in the slurry are allowed to pass through the screen openings, then solidified (cake) layers that form in a compacted state on the inside of the screen from particles of indeterminate form and relatively large size will not cause significant clogging to occur.

[0008] The present invention has been perfected based of the above findings. The main point of the present invention lies in a solid-liquid separation method, characterized in that a slurry containing particles of indeterminate form as solids is continuously supplied to a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation is performed, wherein a screen with an opening size that allows the passage of an amount of solids equivalent to 1 to 10 wt% in the supplied slurry is used as the screen of the screen-type centrifugal separator.

[0009]

[Embodiments of the Invention] The present invention is described in detail hereunder.

[0010] First, the slurry will be described. The slurry used in the present invention contains particles of indeterminate form as solids. A specific example thereof is a slurry containing crude terephthalic acid crystals recovered from a step in which terephthalic acid is manufactured as a result of subjecting *p*-xylene to liquid-phase oxidation, as previously described. This slurry is usually composed of acetic acid as a reaction solvent, terephthalic acid crystals, a catalyst dissolved in the reaction solvent, unreacted starting material, by-products, unprecipitated terephthalic acid, and the like. The average particle size of the terephthalic acid crystals is usually 120 μm ± 40 μm.

[0011] Next, the screen-type centrifugal separator with an internally disposed screw conveyor will be described. In the present invention, it is possible to use screen-type centrifugal separators

having a variety of structures, regardless of their descriptive name, as long as solid-liquid separation can be performed using a screen via the action of a centrifugal force while the material to be treated is transported with a screw conveyor. Examples known in the art include a "decanter-type centrifugal separator" (JP (Kokai) 7-155643, et al.) and a "screen-bowl decanter centrifugal separator" (JP (Kokai) 2000-350946, WO 98/18750, et al.).

[0012] FIG. 1 is a cross-sectional descriptive diagram of a suitable screen-type centrifugal separator that can be used in the present invention. The screen-type centrifugal separator shown in this diagram is constituted from an outer rotating cylinder (1), a screw conveyer (2) that is mounted inside the outer rotating cylinder while allowed to rotate in relative fashion and that comprises a cylindrical rotating shaft (21) and a screw (22), and a slurry supply pipe (3) that is disposed within the rotating shaft of the screw conveyor and that supplies a slurry to the interior of the shaft. The proximal end side of the screw conveyor (2) is provided with a slurry supply port (23) for supplying the slurry to the outer rotating cylinder (1). The outer rotating cylinder (1) is sequentially constituted from a large-diameter region (11) on the proximal end side, a slanted region (12) of a gradually decreasing diameter, and a small-diameter region (13) on which a screen (13a) is provided. An overflow port (4) is provided to the proximal end of the large-diameter region (11), and a solids discharge port (5) is provided to the distal end of the small-diameter region (13).

[0013] In a preferred embodiment, the interior of the rotating shaft (21) of the screw conveyor is partitioned into a slurry supply region (21a) on the proximal end side and a cleaning fluid supply region (21b) on the distal end side. The slurry supply region (21a) is designed to be capable of supplying the slurry via the slurry supply pipe (3), and the cleaning fluid supply region (21b) is designed to be capable of supplying the cleaning fluid via a cleaning fluid supply pipe (6) inserted into the interior of the slurry supply region (3). A cleaning fluid supply port (24) through which cleaning fluid is supplied to the small-diameter region (13) of the outer rotating cylinder (1) is provided on the distal end side of the screw conveyor (2).

[0014] A plurality of openings (71c) are provided to the outer peripheral region of a flange (71) of a drive shaft (7) for the outer rotating cylinder, and these openings constitute the solids discharge port (5). A plurality of openings (81c) are provided to the outer peripheral region of a flange (81) of a drive shaft (8) for the screw conveyor to constitute the overflow port (4).

[0015] All of these elements are housed in a casing (9). The inside of the casing (9) is divided by partitioning walls into an overflow liquid reservoir (91), a filtrate reservoir (92), and a solids reservoir (93); and the filtrate reservoir (92) is further divided by partitioning walls into three compartments (92a), (92b), and (92c) along the longitudinal direction of the small-diameter region (13) for the convenience of filtrate sampling. Discharge piping is provided respectively for the overflow liquid reservoir (91); the compartments (92a), (92b), and (92c) of the filtrate reservoir (92); and the solids reservoir (93).

[0016] An outstanding feature of the present invention is that when solid-liquid separation is performed as a result of continuously supplying a slurry of the above description to a screen-type centrifugal separator of the above description, a screen with an opening size that allows an amount of solids equivalent to 1 to 10 wt% in the supplied slurry to pass therethrough is used as the screen of the screen-type centrifugal separator.

[0017] In other words, the openings in the screen used in the present invention must be of a size capable of allowing continuous passage of, e.g., 1 to 10 wt% (preferably 1.5 to 8 wt%) of the solids (indicated as a (kg/hr)) in the slurry continuously supplied at a rate of A (kg/hr). The passage of particles through the screen openings is believed to occur preferentially with those of indeterminate form and small diameters; in the present invention, therefore, particles of indeterminate form and relatively small diameters in the slurry are allowed to pass through the screen openings.

[0018] Consequently, with the present invention, the solidified layers (cake layers) formed in a compacted state in the inside of the screen, and specifically in the spaces between the outer rotating cylinder (1) and the screw (22), are constituted from particles of indeterminate form and relatively large size. The reason that excessive clogging does not occur with such solidified (cake) layers has yet to be definitively clarified, but the explanation is presumed to be as provided hereunder.

[0019] In other words, when particles of indeterminate form and relatively small size are contained in the solidified (cake) layers, such cakes typically become firm and solid due to a strong bridging action caused as a result. On the other hand, solidified (cake) layers composed of particles of indeterminate form and of relatively large size tend to collapse readily due to the absence of a strong bridging action. Consequently, the solidified (cake) layers in the present

invention are presumed to continuously collapse and rearrange, and are accordingly not believed to cause excessive screen clogging.

[0020] The clogging preventive effect cannot be achieved with screens having an opening size that causes the pass rate to be less than 1 wt%. On the other hand, with screens whose opening size allows more than 10 wt% of the particles to pass, the anticlogging effect may reach saturation while the pass rate becomes too high, thereby resulting in an inefficient operation.

[0021] In the present invention, solid-liquid separation is performed as follows. A slurry is supplied from the slurry supply pipe (3) to the large-diameter region (11) of the outer rotating cylinder (1) via the slurry supply region (21a). The solids and liquids therein are then separated as a result of the centrifugal action of the outer rotating cylinder (1) rotating at a high speed.

[0022] The separated liquid is extracted from the overflow port (4) via the overflow liquid reservoir (91). Meanwhile, the solids are transferred from the slanted region (12) to the small-diameter region (13) by the screw (22). The mother liquor in the solids is also separated as a result of the centrifugal force through the screen (13a). The cleaning fluid is simultaneously supplied from the cleaning fluid supply pipe (6). The cleaning fluid is sprayed on the moving solids through the cleaning fluid supply port (24) via the cleaning fluid supply region (21b). The washed and dehydrated solids are extracted from the solids discharge port (5) via the solids reservoir (93).

[0023]

[Working Examples] The present invention is described in detail below with reference to working examples; however, the present invention shall not be construed to be limited to these examples, and may be embodied in other forms within the purview of the invention.

[0024] Working Example 1 and Comparative Example 1

A slurry containing terephthalic acid crystals (average particle size: 100 μm) obtained in a terephthalic acid manufacturing step was continuously supplied to the screen-type centrifugal separator shown in FIG. 1 furnished with screens having the opening sizes shown in the following Table 1, and the solids and liquids were separated. The centrifugal force on the screen was 750 G. The results are shown in Table 1.

[0025]

[Table 1]

	Screen openings (μm)	Ratio of solids passing through screen openings to solids in supplied slurry (wt%)	Mean particle size of solids passing through screen openings (μm)	Liquid content of recovered solids (wt%)
Working Example 1	100	3	60	11
Comparative Example 1	70	0.5	15	20

[0026] As is clear from the results shown in Table 1, the solids recovered in Working Example 1 have a lower liquid content than those in Comparative Example 1. This is attributable to the fact that clogging was prevented, which allowed the liquid to be satisfactorily removed.

[0027]

[Effect of the Invention] As has been described in the foregoing, the present invention provides a solid-liquid separation method that has been improved so that clogging is prevented in a screen-type centrifugal separator with an internally disposed screw conveyor, and solid-liquid separation can be performed efficiently. Therefore, the present invention is of high commercial value.

[Brief Description of the Drawings]

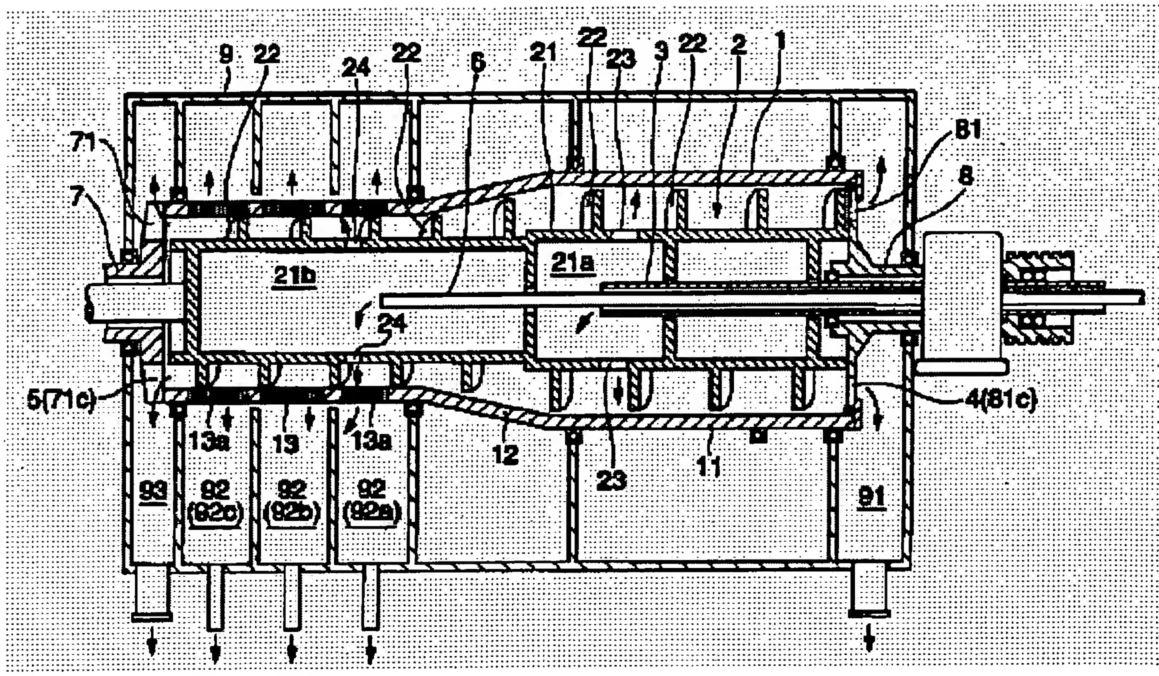
[Figure 1] Cross-sectional illustrative diagram of a screen-type centrifugal separator that is suited for use in the present invention.

[Key]

1	Outer rotating cylinder
11	Large-diameter region
12	Slanted region
13	Small-diameter region
13a	Screen
2	Screw conveyer
21	Rotating shaft
21a	Slurry supply region

21b	Cleaning fluid supply region
22	Screw
23	Slurry supply port
24	Cleaning fluid supply port
3	Slurry supply pipe
4	Overflow port
5	Solids discharge port
6	Cleaning fluid supply pipe
7	Outer rotating cylinder drive shaft
71	Flange
71c	Opening
8	Screw conveyer drive shaft
81	Flange
81c	Opening
9	Casing
91	Overflow liquid reservoir
92	Filtrate reservoir
93	Solids reservoir

[FIG. 1]



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